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(54) COMPOSITIONS FOR REGULATING PLANT GROWTH

(71) We, BASF AKTIENGESELLSCHAFT, a German Joint Stock Company of 6700 Ludwigshafen, Federal Republic of Germany, do hereby declare the invention, for which we pray that a Patent may be granted to us, and the method by which i is to be performed, to be particularly described in and by the following Statement:–

The present invention relates to compositions for regulating plant growth, which contain a mixture of active ingredients, and to processes for regulating plant growth by means of these compositions.

The use of certain quaternary ammonium compounds, salts of N,N-disubstituted heterocyclic amines, e.g. N,N-dimethylpiperidinium salts (our British Patent Specification No 1 414 259) or of certain phosphonic acid derivatives, e.g. 2-chloroethane-phosphonic acid (British Patent Specification No. 1 194 433) for regulating plant growth has been disclosed. We have also disclosed and claimed mixtures of (a) certain trimethylammonium salts, e.g. N,N,N-trimethyl-N-2-chloroethylammonium chloride, or dimethylhydrazinium salts or dimethylmorpholinium salts and (b) certain phosphonic acid compounds, e.g. 2-chloroethanephosphonic acid, as well as their use for regulating plant growth (our British Patent Specification No. 1 483 915). We have also disclosed and claimed salts of certain quaternary ammonium compounds and phosphonic acids, e.g. the N,N-dimethylpiperidinium salt of 2-chloroethanephosphonic acid, and their use for regulating plant growth (our British Patent Specification No. 1 499 189).

We have found that mixtures of

a) a quaternary substituted thianium or ammonium salt selected from N,N-dimethylazacycloheptanium salts, N,N-dimethylpiperidinium salts, N,N-dimethyl-hexahydropyridazinium salts, N,N-dimethyl-tetrahydropyridazinium salts, N-methyl-pyridinium salts, N,N-dimethyl-pyrrolidinium salts, and S-methylthiacyclohexanium salts, each with non-phytotoxic anions

b) a phosphonic acid compound selected from 2-chloroethylphosphonic acid, 2-chloroethyl-0-(2′-aminoethyl)-phosphonic acid, 2-chloroethyl-0-(2′-amino-n-butyl-phosphonic acid, 2-chloroethylphosphonic acid, N,N-dimethylamide, 2-chloroethylphosphonic acid N-methylamide, vinylphosphonic acid, propylphosphonic acid, phosphonomethylglycine, bis-phosphonomethylglycine and benzyl-phosphonic acid, are very useful for regulating growth.

The salts are essentially any salts with non-phytotoxic anions, for example anions disclosed in the specifications mentioned above. Preferred salts are the halides, e.g. the bromides and particularly the chlorides.

Regulating plant growth may entail, for example, any one or more of the following changes:

inhibition of lengthening of the cells, for example shortening of the stems and internodal distances, strengthening of the stem wall and, as a result, improvement in resistance to lodging, as a precondition for ensuring good yields of cereals and other graminaceous plants for use as seed, or good yields of fibrous plants for the production of textile fibers;

compact growth in ornamentals to ensure economical production of improved quality plants;

promotion of fruiting, for example increased fruit set in the case of pomes, drupes and aggregate fruit, e.g. grapes, citrus fruit, almonds, olives, cocoa and coffee;

deliberate sex differentiation, with the object of increasing the yield, for example in the case of Cucurbitaceae and papaya;

promotion of deliberate senescense, with the object of causing abscission, for example stimulating the loosening of fruit, so as to facilitate the mechanical harvesting of citrus fruit, pomes, drupes and aggregate fruit, olives, almonds, coffee and indehiscent fruits;

defoliation of nursery-grown trees and ornamentals for despatch in the autumn;

defoliation of trees to break parasitic infection chains, for example Gloeosporium heveae in Heva brasiliensis; and

promotion of ripening, for example in tomatoes, citrus fruit, pineapples and coffee, with the object of being able to programme harvesting and promoting fruit color or, in the case of cotton, with the object of concentrating the harvest into 1 or 2 pickings, and breaking the nutritional chain for harmful insects.

The mixtures according to the invention exhibit a synergistic action, especially in cereals,

i.e. the action of the mixture is greater than the sum of the actions of the individual active ingredients. Furthermore, the mixtures are better tolerated by plants than are conventional active ingredients, and improve the stability of cereal stems, so that less lodging of the plants is observed.

Preferably, component (a) contains quaternary nitrogen and component (b) contains halogen. In particular, mixtures of N,N-dimethylpiperidinium chloride or N,N-dimethylhexahydropyridazinium bromide (or salts of these cations with other non-phytotoxic inorganic anions), particularly with 2-chloroethylphosphonic acid, 2-chloroethyl-0-(2'-aminoethyl)-phosphonic acid, 2-chloroethylphosphonic acid N,N-dimethylamide or 2-chloroethyl-0-(2'-amino-n-butyl)-phosphonic acid, exhibit a good growth-regulating action.

The ratio in which the active ingredients (a) and (b) are mixed may vary within wide limits and may be, for example, from 10:1 to 1:10 by weight, preferably from 4:1 to 1.3:1. Molar ratios of from 5:1 to 10:9 and 9:10 to 1:5 may be mentioned. Normally it is preferred for the cation of component (a) to be present in excess of the stoicchiometrically equivalent amount of component (b), although lesser amounts may be used.

The application of the compositions according to the invention may be effected, for instance, in the form of directly sprayable solutions, suspensions, including high-strength aqueous suspensions, or emulsions, by spraying, atomizing or watering. The forms of application depend on the purpose for which the agents are being used; in any case they should ensure a very fine distribution of the compositions according to the invention. Preferably, however, the active ingredients are employed in the form of aqueous solutions which may be obtained by diluting aqueous concentrates with water.

Aqueous formulations may be prepared from aqueous concentrates or emulsion concentrates by adding water. To prepare emulsions, the ingredients as such may be homogenized in water by means of wetting agents or dispersants, adherents or emulsifiers. However, concentrates which are suitable for dilution with water may also be prepared from the active ingredient, possibly a wetting agent, adherent, dispersant or emulsifying agent and water.

Examples of suitable surfactants are: alkali metal salts, alkaline earth metal salts and ammonium salts or ligninsulfonic acid, naphthalenesulfonic acid and phenolsulfonic acids, alkylarylsulfonates, alkyl sulfates, alkylsulfonates, alkali metal salts and alkaline earth metal salts of dibutylnaphthalenesulfonic acid, lauryl ether-sulfate and fatty alcohol-sulfates, alkali metal salts and alkaline earth metal salts of fatty acids, salts of sulfated hexadecanols, heptadecanols and octadecanols, salts of sulfated fatty alcohol glycol ethers, condensation products of sulfonated naphthalene derivatives with formaldehyde, condensation products of naphthalene or of naphthalenesulfonic acids with phenol and formaldehyde, polyoxyethylene octylphenol ethers, oxyethylated isooctylphenol, oxyethylated octylphenol and oxyethylated nonylphenol, alkylphanol polyglycol ethers, tributylphenyl polyglycol ethers, alkylaryl polyether alcohols, isotridecyl alcohol, fatty alcohol/ethylene oxide condensates, oxyethylated castor oil, polyoxyethylene alkyl ethers, oxyethylated polyoxypropylene, lauryl alcohol polyglycol ether acetal, sorbitol esters, lignin, sulfite waste liquors and methylcellulose.

Preferred formulations are, as stated above, aqueous solutions. These will normally be free from organic solvents and solid fillers or diluents and we have found that it is not necessary for a wetting agent, dispersant, emulsifier or adherent to be present. Compositions according to the invention can therefore be free or largely free from metal or ammonium cations and will in particular not contain such cations to the extent arising from the provision of component (b) as a metal or salt, i.e. stoichiometrically equivalent amounts of such cations and the characterizing anions of component (b). Hence, preferred formulations are aqueous solutions containing as substantially the only ionic species, (a) stoichiometrically equivalent amounts of a characterizing cation of component (a), e.g. the N,N-dimethyl-piperidinium cation, and the non-phytotoxic anion of its salt, e.g. the chloride anion, and (b) a preferably less than stoichiometrically equivalent amount of a characterizing anion of component (b), although greater amounts can be used.

The compositions can be manufactured by more admixture of the active ingredients and this will be conducted under conditions where the active ingredients do not react completely with one another, if at all. This mixing preferably occurs in one aqueous medium free from other additives and a very useful composition is obtained by mixing from 1.3 to 4 parts by weight of N,N-dimethylpiperidinium chloride with 1 part by weight of 2-chloroethylphosphonic acid in the presence of sufficient water to provide a solution containing at least 100 g of the mixture per liter. This concentrate can then be diluted with water for use.

The formulations generally contain from 0.1 to 95 per cent by weight of active ingredient, preferably from 0.5 to 90 per cent by weight. A convenient retail formulation is an aqueous concentrate containing at least 100 g/l of the mixture, whereas for application the formulation will conveniently contain 0.5 to 10 g/l of the mixture.

Further, oils of various types, herbicides, fungicides, nematocides, insecticides, bactericides, trace elements, fertilizers, surfactants, synergistic agents, anti-foam agents (e.g. silicones), growth regulators or antidotes may be added to the mixtures.

In use the compositions according to the invention are applied to the plants whose growth is to be regulated or to the locus in which such plants are growing. The compositions are of particular importance for cereal growth regulation, especially for barley, but also find use in other areas, e.g. potatoes, sugar beat, ornamentals or fruiting plants, such as are mentioned above. An application rate of from 200 g to 2 kg of mixture per hectare may be mentioned.

The Examples of field tests, given below, confirm the fact that the biological action of the mixture is greater than that of the individual active ingredients.

EXAMPLE 1

Plant: spring barley, "Villa" variety.

The treatment with the aqueous solutions of the active ingredients was carried out 74 days after sowing. 101 days after sowing, the height to which the plants had grown was measured (in each case using the average value from 100 measurements).

The resistance to lodging of the plants was assessed 124 days after sowing, with figures of merit ranging from 1 for the greatest resistance to 9 for no resistance.

Active ingredient	Amount applied g/ha	Growth height cm	Reduction in growth cm	Resistance to lodging	Improvement in resistance to lodging
Untreated	–	85.5		3.5	
DPC	920	83.4	2.1	2.5	1.0
CEPA	442	80.8	4.7	2.5	1.0
DPC + CEPA	920 + 442	77.4	8.1	1.0	2.5
DPC + CEPA	920 + 884	76.4	9.1		

DPC = N,N-dimethylpiperidinium chloride
CEPA = 2-chloroethylphosphonic acid.

The above results confirm that the mixture exhibits a synergistic action, compared with the individual components, as far as reduction in growth height is concerned.

EXAMPLE 2

Plant: winter barley, "Birgit" variety.

Treatment was carried out with the aqueous solutions of the active ingredient 219 days after sowing. The barley was harvested 281 days after sowing.

Active ingredient	Amount applied g/ha	Grain yield dt/ha	%
Untreated	–	72.4	100
DPC	920	72.5	100
CEPA	442	78.3	108
DPC + CEPA	920 + 442	80.0	110

This Example demonstrates that the mixture exhibits synergism and produces an increase in yield compared with the individual components.

EXAMPLE 3

Plant: oats, "Flämingskrone" variety.

Treatment was carried out with the aqueous solutions of the active ingredients 72 days after sowing. Resistance to lodging was assessed 131 days after sowing.

Active ingredient	Amount applied g/ha	Resistance to lodging	Improvement in resistance to lodging
Untreated	–	7.0	
DPC	460	6.7	0.3
CEPA	221	7.0	0
DPC + CEPA	460 + 221	63.	0.7

Even when the untreated plants are more severely lodged, the mixture exhibits a synergistic

improvement in resistance lodging.

EXAMPLE 4

Plant: oats, "Borrus" variety.

Treatment with the aqueous solutions of the active ingredients was carried out 80 days after sowing. The growth height was measured 105 days after sowing. The oats were harvested 4 months after sowing.

Active ingredient	Amount applied g/ha	Growth height cm	Reduction in growth height cm	Grain dt/ha	yield %
Untreated	—	83.9		42.1	100
DPC	460	83.9	0	38.3	91
CEPA	221	83.4	0.5	38.1	90
DPC + CEPA	460 + 221	83.1	0.8	45.2	107
DPC + CEPA	460 + 442	83.0	0.9	40.8	97

The results of this experiment clearly show the synergistic action of the mixtures, relative to the individual components.

EXAMPLE 5

Plant: winter rye, "Carokurz" variety.

Treatment with the aqueous solutions of the active ingredients was carried out 196 days after sowing. The growth height was measured 235 days after sowing.

Active ingredient	Amount applied g/ha	Growth height cm	Reduction in growth height, cm
Untreated	—	142.7	
DPC	460	140.2	2.5
CEPA	221	141.0	1.7
DPC + CEPA	460 + 221	134.5	8.2

As with the other varieties of cereals, the mixture here again leads to a synergistic reduction in growth height.

EXAMPLE 6

Plant: winter rye, "Kustro" variety.

Treatment with the aqueous solutions of the active ingredients was carried out 209 days after sowing. Harvesting took place 282 days after sowing.

Active ingredient	Amount applied g/ha	Grain yield dt/ha	%
Untreated	—	40.5	100
DPC	1,380	42.9	106
CEPA	1,440	38.6	95
DPC + CEPA	690 + 240	43.9	108

This Example shows that a mixture in the ratio of about 3:1 has a beneficial effect on the grain yield.

EXAMPLE 7

Plant: Indian corn, "Limac" variety.

Treatment with the aqueous solutions of the active ingredients was carried out 60 days after sowing. The growth height was measured 90 days after sowing. Harvesting took place 167 days after sowing.

Active ingredient	Amount applied g/ha	Growth height cm	Reduction in growth height, cm	Cobs/cm² number	%
Untreated	—	168.0		9.1	100
DPC	920	164.6	3.4	9.2	101
CEPA	721	136.1	31.9	8.9	98
DPC + CEPA	920 + 721	129.4	38.6	9.4	103

The mixture shows a synergistic action, in respect of reduction in growth height and cob count, relative to the individual components. Excessive growth of the plants, which is generally undesirable, is restricted, to the benefit of an increase in yield.

EXAMPLE 8

Plant: winter barley. "Mirra" variety.

Treatment with the aqueous solutions of the active ingredients was carried out 200 days after sowing. The growth height was measured 227 days after sowing. The resistance to lodging was

assessed 270 days after sowing.

Active ingredient	Amount applied g/ha	Growth height cm	Reduction in growth height cm	Resistance to lodging	Improvement
1) Untreated	–	94.0		6.5	0
2) N,N-dimethyl-hexa-hydro-pyridazinium bromode	920	90.2	3.8	4.0	2.5
3) 2-Chloroethyl-0-(2'-amino-n-butyl)-phosphonic acid	960	89.3	4.7	4.5	2.0
2 + 3	920 + 480	78.0	16.0	1.5	5.0

The mixture exhibits a synergistic action, in respect of reduction in growth height and improved resistance to lodging, relative to the individual components.

EXAMPLE 9

Plant: winter barley, "Ogra" variety.

Treatment with the aqueous solutions of the active ingredients was carried out 213 days after sowing. The growth height and the resistance to lodging were evaluated respectively 232 and 288 days after sowing. Harvesting took place 244 days after sowing.

Active ingredient	Amount applied, g/ha	Growth height, cm	Reduction in growth height, cm	Resistance to lodging	Improvement in resistance to lodging	Grain yield dt/ha	Grain yield %
1) Untreated	–	129.0		4.0		58.1	100
2) DPC	920	126.4	2.6	3.7	0.3	57.5	99
3) 2-Chloro-ethyl-0-(2'-amino-ethyl)-phos-phonic acid	480	127.7	1.3	2.7	1.3	59.5	102
4) 2 + 3	920 + 480	122.6	6.4	1.3	2.7	60.9	105

The mixture again exhibits a synergistic effect relative to the individual components. The growth height, the resistance to lodging and the grain yield are substantially improved.

EXAMPLE 10

Plant: winter barley, "Dura" variety.

Treatment with the aqueous solutions of the active ingredients was carried out 124 days after sowing. Harvesting took place 185 days after sowing.

Active ingredient	Amount applied g/ha	Grain yield dt/ha	Grain yield %
1) Untreated	–	47.5	100
2) DPC	920	51.4	108
3) 2-Chloroethylphosphonic acid N,N-dimethylamide	240	49.1	103
4) 2 + 3	920 + 480	53.7	113

The Example illustrates the considerable increase in yield achieved with the mixture.

EXAMPLE 11

Spring rye, "Beacon" variety.

Treatment with the aqueous solutions of the active ingredients was carried out 59 days after sowing. The growth height was measured 100 days after sowing.

Active ingredient	Amount applied g/ha	Growth height cm	Reduction in growth height, cm
1. Untreated	–	66	
2. DPC	515	65	1
3. CEPA	134	67	–1
4. 2+3	515 + 134	62	4

This Example shows that even using a mixture in the ratio of 4:1, a synergistic effect is achieved, relative to the individual components.

EXAMPLE 12

Plant: winter rye, "Kustro" variety.

Treatment with the aqueous solutions of the active ingredients was carried out 204 days after sowing. The growth height was measured after 232 days and harvesting took place 285 days after sowing.

Active ingredient	Amount applied g/ha	Growth height, cm	Reduction in growth height cm	Grain Yield dt/ha	%
1) Untreated	–	154.2		55.70	100
2) A	1,840	143.2	11.0	55.02	99
3) DPC + CEPA	1,200 + 620	134.8	19.4	56.74	102

A = N,N-dimethylpiperidinium salt of 2-chloroethanephosphonic acid (known from German Laid-Open Application DOS 2,422,807).

The reduction in growth height and the increased grain yield demonstrate the substantially superior action of mixture 3, relative to the known salt A.

EXAMPLE 13

Plant: winter rye, "Kustro" variety.

Treatment with the aqueous solutions of the active ingredients was carried out 206 days after harvesting. The resistance to lodging was assessed 261 days after sowing, and harvesting took place 288 days after sowing.

Active	Amount applied g/ha	Resistance to lodging	Improvement in resistance lodging	Grain yield dt/ha	%
1) Untreated	–	6.8		53.1	100
2) A	1,380	3.8	3.0	54.6	103
3) DPC+CEPA	915 + 465	2.3	4.5	56.4	106

A = N,N-dimethylpiperidinium salt of 2-chloroethanephosphonic acid.

Here again, mixture 3 is substantially superior to salt A in respect of improvement in resistance to lodging, and increase in yield.

EXAMPLE 14

Plant: spring rye, "Somro" variety.

Treatment with the aqueous solutions of the active ingredients was carried out 85 days after sowing, the growth height was measured 100 days after sowing and harvesting took place 153 days after sowing.

Active ingredient	Amount applied, g/ha	Growth height, cm	Reduction in growth height, cm	Grain yield dt/ha	%
1) Untreated	–	196.3		34.4	100
2) A	920	184.9	11.4	34.0	99
3) DPC+CEPA	610+310	183.4	12.9	36.1	105

A = N,N-dimethylpiperidinium salt of 2-chloroethanephosphonic acid.

The reduction in growth height and the increased grain yield show that in the case of spring rye the action of mixture 3 is again superior to that of salt A.

EXAMPLE 15

Plant: winter barley, "Ago" variety.

Treatment with the aqueous solutions of the active ingredients was carried out 208 days after sowing. The resistance to lodging was assessed 271 days after sowing and harvesting took place 279 days after sowing.

Active ingredient	Amount applied, g/ha	Resistance to lodging	Improvement in resistance to lodging	Grain yield dt/ha	%
1) Untreated	–	7.5		58.6	100
2) A	1,840	7.0	0.5	50.5	86
3) DPC + CEPA	1,220+620	5.0	2.5	64.5	110

A = N,N-dimethylpiperidinium salt of 2-chloroethanephosphonic acid.

Compared to treatment with salt A, treatment with mixture 3 results in a substantially higher resistance to lodging, whilst the better toleration of mixture 3 by the crop results in a substantial increase in yield.

WHAT WE CLAIM IS:–

1. A composition for regulating plant growth, comprising a mixture of

a) a quaternary substituted thianium or ammonium salt selected from N,N-dimethyl-azacycloheptanium salts, N,N-dimethyl-piperidinium salts, N,N-dimethyl-hexahydro-pyridazinium salts, N,N-dimethyl-tetrahydro-pyridazinium salts, N-methyl-pyridinium salts, N,N-dimethyl-pyrrolidinium salts, and S-methyl-thiacyclohexanium salts, each with non-phytotoxic anions, and

b) a phosphonic acid compound selected from 2-chloroethylphosphonic acid, 2-chloroethyl-0-(2'-aminoethyl)-phosphonic acid, 2-chloroethyl-0-(2'-amino-n-butyl)-phosphonic acid, 2-chloroethylphosphonic acid N,N-dimethylamide, 2-chloroethylphosphonic acid N-methylamide, vinylphosphonic acid, prophylphosphonic acid, phosphonomethylglycine, bis-phosphonomethylglycine and benzylphosphonic acid.

2. A composition as claimed in Claim 1 wherein component (b) of the mixture is a

halogen-containing compound and component (a) of the mixture is a quaternary nitrogen-containing compound.

3. A composition as claimed in any of Claim 1 of 2 wherein component (a) of the mixture is an N,N-dimethylpiperidinium or N,N-dimethylhexahydropyridazinium salt of an inorganic non-phytotoxic anion.

4. A composition as claimed in any of Claims 1 to 3 wherein component (b) of the mixture is 2-chloroethylphosphonic acid.

5. A composition as claimed in Claim 1, which comprises a mixture of N,N-dimethylpiperidinium chloride and 2-chloroethylphosphonic acid.

6. A composition as claimed in any of Claims 1 to 5 wherein the weight ratio of component (a) to component (b) is from 10:1 to 1:10.

7. A composition as claimed in Claim 6 wherein the molar ratio of component (a) to component (b) is from 5:1 to 10:9.

8. A composition as claimed in Claim 6 wherein the molar ratio of component (a) to component (b) is from 9:10 to 1:5.

9. A composition as claimed in Claim 5 wherein the weight ratio of component (a) to component (b) is from 4:1 to 1.3:1.

10. A composition as claimed in any of Claims 1 to 9, wherein the active ingredients are present in aqueous solution.

11. A composition as claimed in Claim 10 wherein the aqueous solution is largely free from metal or ammonium cations and free from organic solvents and solid diluents or fillers.

12. A composition as claimed in Claim 11 wherein the aqueous solution is free from wetting agents, dispersants, emulsifiers and adherents.

13. A composition as claimed in Claim 10 consisting of an aqueous medium containing, as substantially the only ionic species, (a) stoichiometrically equivalent amounts of N,N-dimethylpiperidinium cations and chloride anions and (b) a greater or lesser amount (on an equivalent basis) of 2-chloroethylphosphate anions.

14. A composition as claimed in Claim 1 and substantially as hereinbefore specifically described or as exemplified.

15. A process for the manufacture of a composition as claimed in Claim 1 which comprises mixing component (a) as defined in any of Claims 1 to 3 and component (b) as defined in any of Claims 1, 2 and 4 under conditions such that the components do not react together completely if at all.

16. A process as claimed in Claim 15 where the components are mixed in an aqueous medium free from other additives.

17. A process as claimed in Claim 16 wherein from 1.3 to 4 parts by weight of N,N-dimethylpiperidinium chloride are mixed with 1 part by weight of 2-chloroethylphosphonic acid in the presence of sufficient water to provide a solution containing at least 100 g of the mixture per liter of solution, and the solution is subsequently diluted with water.

18. A composition as claimed in Claim 1 when manufactured by a process as claimed in any of Claims 15 to 17.

19. A composition as claimed in any of Claims 10 to 14 or 18 in the form of an aqueous concentrate containing at least 100 g/l of active ingredients.

20. A composition as claimed in any of Claims 10 to 14 in the form of a dilute aqueous sprayable solution containing from 0.5 to 10 g/l of active ingredients.

21. A process for regulating plant growth wherein a composition as claimed in any of Claims 1 to 14, 18 or 20 is applied to the plants or to the locus in which the plants are growing.

22. A process as claimed in Claim 21 wherein the plants are cereals, potatoes, sugar beet, ornamentals or fruiting plants.

23. A process as claimed in Claim 22 wherein the plants are barley plants.

24. A process as claimed in any of Claims 21 to 23 wherein the composition is applied at a rate of from 200 g to 2 kg of active ingredient per hectare.

25. A process as claimed in Claim 21 and substantially as hereinbefore specifically described.

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